

LIST OF U.S. CUSTOMS LABORATORY METHODS

USCL NUMBER	METHOD	TITLE
64-01	USCL Manual NHM	<u>Footwear</u>

U.S. CUSTOMS LABORATORY METHODS

USCL METHOD 64-01

INDEX

Footwear Analysis: External Surface Area of Upper by Planimeter and Image Analysis; Identity of Footwear Components; Footwear Construction; Weight Percent of Footwear Components

Conditioning and Testing.

SAFETY PRECAUTIONS

This method does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this method to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to its use.

1 SCOPE

This method specifies procedures for the following; percent of the external surface area of upper materials; identity of footwear components; method of construction; and weight percent of components.

2 FIELD OF APPLICATION

This method is suitable for footwear contained in Chapter 64 of the Harmonized Tariff Schedule.

3 REFERENCES

ISO 139

Textiles - Standard Atmospheres for

4 PRINCIPLE

By incorporating the reference methods used by the three countries, a standard Harmonized NAFTA method can be used for the analysis of footwear.

5 APPARATUS AND REAGENTS

5.1 Surface Area Measuring Apparatus

5.1.1 Polar Compensating Planimeter (Manual or Electronic)

The instrument shall have: a measuring mechanism incorporating a measuring wheel with a vernier and a counting dial; a pole weight; a fixed pole arm; a fixed tracer arm; and a test/calibration ruler. The instrument shall have an accuracy of $\pm 0.2\%$ or better.

5.2 Image Analyzer

A system consisting of a camera system (optional), digitizing board, and a data system. The combined system shall be capable of meeting the same specification for precision and accuracy as above for the polar

compensating planimeter.

5.3 Compound Optical Microscope

The instrument shall have a resolving power sufficient to establish the morphological characteristics of footwear components such as leather, wool and other animal fibers, vegetable fibers and materials, and man-made materials.

5.4 Infrared Spectrophotometer

The instrument shall have the specifications necessary to identify such footwear components as synthetic plastics materials and rubber.

5.5 Analytical Balance

The balance shall have the sensitivity of three (3) decimal places.

5.6 Photocopying Equipment

The photocopier shall be of a type capable of producing sharp images of relatively thick footwear components, specifically upper materials.

5.7 Other Apparatus and Reagents

The method requires various reagent-grade chemicals such as:

- a) acetone
- b) chloroform
- c) tetrahydrofuran (THF)
- d) alcoholic potassium hydroxide solution
- e) alcohol (ethanol, ETOH)

and common laboratory equipment

such as:

- f) razor/utility knives
- g) autopsy saw
- h) magnifying glasses
- i) instrument accessories
- j) glassware, etc.

5.8 Conditioning and testing atmosphere

Weight determination should be carried out in the standard temperate atmosphere for testing, according to the Explanatory Notes to Section XI of the Harmonized Tariff Schedule. Other analyses may be carried out in the ambient atmosphere.

6 PREPARATION OF THE SAMPLE

6.1 Examination

Examine the footwear and, as far as is practicable by observation, note:

1. Identifying marks, labels, sizes, etc.
2. The style (sandal, rainwear, athletic shoe, etc).
3. Each type of upper material.
4. Type of the sole material.
5. The method of construction.
6. Any unusual characteristics (eg., considerable ornamental stitching, etc.)

6.2 Determination of Required Analyses

Based on these observations and conclusions, determine which of the analyses described in Section III, below, will be necessary to permit a proper classification of the footwear

sample. Note that in many cases several different analyses may be indicated. These should be performed in a sequence such that one analysis does not preclude or nullify the results of any subsequent analyses (eg., do not destroy a portion of the upper material in an identification analysis if the external surface area of that material must be determined).

7 PROCEDURE FOR THE DETERMINATION OF EXTERNAL SURFACE AREA(S) OF THE UPPER BY PLANIMETER AND IMAGE ANALYSIS

7.1 Procedure

7.1.1 Identify all external surfaces of the upper to be included in the determination, taking note of all applicable administrative rulings and judicial interpretation.

7.1.2 Using a sharp cutting tool such as a razor knife, or autopsy saw cut the footwear sample to remove the upper, and, if necessary, cut the upper into pieces so that they may be laid flat. It is recommended that the number of cuts be held to a minimum. Where necessary, remove the box toe and the counter.

7.1.3 Place the flat pieces of upper on the surface of a photocopier such that the external surface is against the glass. If the color of the sample is black or dark it would be advisable to outline

the area to be traced on the photocopy with white crayon or paint pen. Obtain photocopy images of the upper pieces. It is recommended that the set of photocopy images be retained.

7.1.4 Determine the relative areas of each of the components with a planimeter, or image analyzing system following the instructions for that instrument. (To determine the areas of large components, it may be necessary to sub-divide them and measure each subdivision separately.) Measurements should be measured to two (2) decimal places. Perform all measurements in duplicate.

7.2 Calculation and expression of results

7.2.1 Add the average values of all area measurements of each type of upper material to produce the subtotal area for each type.

7.2.2 Add the average value of all area measurements of all types of upper materials to produce the total area.

7.2.3 Calculate the relative percentage area of each type of upper material by dividing each subtotal area by the total area and multiplying by 100. Two decimals should be carried as such until the final calculations for percent. Relative percentage should be rounded to the nearest 0.1%, and reported as such where necessary for classification.

7.2.4 Example:

Six pieces of upper material are photocopied; three are of plastic, two

are of leather, and one is of textile. The average areas (resulting from duplicate planimeter determinations) are:

Area plastic #1 = 4.50 units
 Area plastic #2 = 4.60 units
 Area plastic #3 = 2.90 units
 Area leather #1 = 5.20 units
 Area leather #2 = 0.80 units
 Area textile #1 = 2.00 units

Then:

Subtotal area of plastic = 12.00 units
 Subtotal area of leather = 6.00 units
Subtotal area of textile = 2.00 units
 Total external surface area = 20.00 units

The relative areas of each type of upper material are calculated as:

Relative area of plastic
 $= \frac{12 \times 100}{20} = 60\%$

Relative area of leather
 $= \frac{6 \times 100}{20} = 30\%$

Relative area of textile
 $= \frac{2 \times 100}{20} = 10\%$

7.3 Notes

7.3.1 Overlay materials

When necessary, calculate both with and without overlay materials.

7.3.2 Regular geometric patterns

7.3.2.1 To determine the area of upper pieces which have been cut in a

regular geometric pattern (such as strips of material with "saw-tooth" edges), it is recommended that, wherever possible, measurements be taken along straight lines.

7.3.2.2 For example, the following are some of the means of measuring the area of commonly encountered "saw-tooth" patterns.

7.3.2.2(a) From the left hand base of the left "tooth" to the apex of the right "tooth";

7.3.2.2(b) Along the "half-height" line;

7.3.2.2(c) Along the base line of one row of "teeth" and along the apex line of the opposite row of teeth.

7.3.2.3 In drawing "equivalent area" lines on images of upper pieces which are not basically "rectangular", exercise caution to ensure that the entire area of the image, but only that area, is measured.

8 PROCEDURE FOR THE OF ANALYSIS FOR IDENTITY OF FOOTWEAR COMPONENTS

8.1 Plastics Materials

For the purposes of this method, "rubber or plastics" materials are those substances provided for by name under HS Chapters 39 (plastics - headings 3901 to 3914) and 40 (rubber). Their identity may be

determined by appropriate chemical, physical, or instrumental procedures such as burning characteristics, solubility tests, spot or color tests, etc.

8.1.1 The following procedure for the identification of polyvinyl chloride (PVC) is representative:

- 8.1.1(a)** Dissolve a suitable portion of the plastic material in tetrahydrofuran (THF) and centrifuge.
- 8.1.1(b)** Decant the supernatant liquid into a clean centrifuge tube.
- 8.1.1(c)** Add 95% ethanol until the PVC precipitates, and centrifuge.
- 8.1.1(d)** Discard the supernatant liquid.
- 8.1.1(e)** Dissolve the precipitate in THF.
- 8.1.1(f)** To prepare a thin film for spectroscopy, pour the solution into a disposable aluminum pan, evaporate the THF, and peel off the film.
- 8.1.1(g)** Alternatively, cast a film from the solution onto a KBr window and take the spectrum.
- 8.1.1(h)** Confirm PVC by reference to standard spectra.

8.2 Rubber

Appropriate procedures should be used to identify rubber materials; the following are representative:

8.2.1 Polychloroprene by Infrared Spectroscopy

Obtain the pyrolysate by appropriate techniques and run a thin-film spectrum between salt plates. The pyrolysate of polychloroprene may yield a variable spectrum, and must be compared with reference spectra.

8.2.2 Natural rubber

- 8.2.2(a)** Color tests
The Weber Reaction and the trichloroacetic acid test are characteristic of polyisoprene.
- 8.2.2(b).** Infrared Spectroscopy
Obtain the pyrolysate by appropriate techniques and run a thin-film spectrum between salt plates. Confirm by reference to standard spectra.

8.2.3 Other Rubber Materials

Appropriate spot and/or color tests may be used, but conclusive identification of other rubber materials should be done by means of infrared spectroscopy (usually on pyrolysates) with reference to pyrolysate spectra.

8.3 Leather¹

For the purposes of this method, "leather" refers to the hide or skin of any animal (including birds or fish) which has been permanently

¹ Harmonized System Explanatory Notes for Section 41,09 on pp 607, 608.

combined with chromium salts, formaldehyde, or any of several other natural or synthetic tanning substances. The following tests may indicate the presence of leather:

8.3.1 Procedures

8.3.1.1 Ignition and observation of burning characteristics.

8.3.1.2 Extraction and identification of common tanning materials by spot or color tests or instrumental techniques.

8.3.1.3 Preparation and microscopical examination with reference to known leather standards;

8.4 Patent Leather

For the purposes of this method, "patent leather" refers to leather which has been finished with drying oil (eg., linseed oil), varnish, or synthetic resins. In addition the term applies to materials produced by overlaying leather with a pre-existing plastic sheet or film. The thickness of the coating or the sheet must not exceed 0.15 mm, or the material would be considered plastic.

Patent laminated leather, also known as patent coated leather, is leather covered with a pre-formed sheet of plastics of a thickness exceeding 0.15 mm but less than half the total thickness and having the lustrous mirror-like appearance of patent leather. These materials are described in chapter 39.

Patent leather and its finish may be identified by appropriate techniques such as those listed above. The thickness of plastic coatings or laminations can be measured microscopically using a micrometer, or comparison to a standard or known thickness.

8.5 Fibers

For the purposes of this method, the term "fibers" means unspun fibrous vegetable materials, vegetable fibers, wool, silk, or other animal fibers, man-made fibers, paper yarns, or any combination thereof.

8.5.1 These fibers may be identified using appropriate instrumental or wet chemical techniques, such as microscopical examination, spectroscopy, thermoanalytical techniques or spot, color, or solubility tests.

9 PROCEDURE FOR THE ANALYSIS OF FOOTWEAR CONSTRUCTION

The various methods of footwear construction are described in Explanatory Notes, Vol. 3 Chapter 64. All applicable administrative rulings and judicial interpretation should also be taken into consideration.

In many instances it will be necessary to cut through a portion of the shoe (usually at the point corresponding to the ball of the foot) in order to determine its method of construction. This cut may best be accomplished by

means of an autopsy saw, electric saw or cutting wheel, but a hacksaw may also be used. The use of a razor knife or similar implement is not recommended for safety considerations.

10 PROCEDURE FOR THE ANALYSIS OF WEIGHT PERCENT OF FOOTWEAR COMPONENTS

10.1 General Procedure

The weight percent composition of a footwear sample is determined as follows:

- 10.1.1** Weigh the footwear on an analytical balance.
- 10.1.2** Identify the footwear materials by appropriate techniques.
- 10.1.3** Disassemble the footwear, and combine like materials such as: rubber, plastic, metal, textile materials, leather, wood, cork, cardboard, etc., using whatever physical or chemical means as may be practicable.
- 10.1.4** Weigh each of the components on an analytical balance after conditioning in a standard temperate atmosphere for testing, as specified in the Explanatory Notes, Section XI (IV).
- 10.1.5** Calculate and report the percent by weight of each type of material to the nearest tenth

percent.

10.2 Formed Uppers

When necessary, determine the complete component breakdown of all upper materials, including fabric blends.

10.3 Components

10.3.1 Rubber

For the purposes of this method, the weight of the "rubber" components includes the weight of all fillers, reinforcements, and rubber-processing chemicals.

10.3.2 Metallic Items

Items of metal such as eyelets, buckles, shanks, toe caps, etc., should be grouped together and weighed. The paint on metal components need not be removed for percent-by-weight determinations.

11 NOTES ON PROCEDURES

- 11.1** The Explanatory Notes to Chapter 64 of the HS should be consulted for further information and followed whenever possible.

12 DOCUMENTATION OF RESULTS

The laboratory worksheet should contain the following particulars:

- a) a statement that the test was performed in accordance with this

Harmonized NAFTA standard;

- b) the method used;
- c) where more than one specimen is tested, the result for each specimen;
and
- d) the date of the test.